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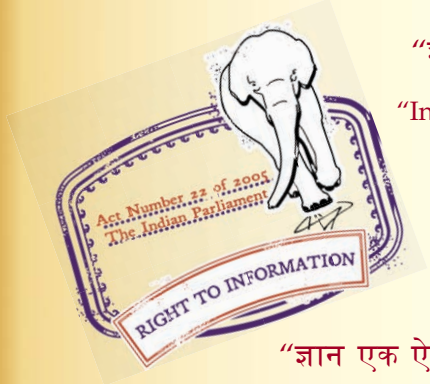

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METHODS OF SAMPLING ROCK PHOSPHATE

UDC 622·364 : 620·113

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NEW DELHI 110002

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September 1985

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Indian Standard

METHODS OF SAMPLING ROCK PHOSPHATE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 4 December 1984, after the draft finalized by the Methods of Sampling Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Rock phosphate is mainly used for the production of chemical fertilizers. Small quantities are also used in the manufacture of elemental yellow phosphorous and phosphorous compounds. The total consumption of rock phosphate in India is estimated to be approximately 2 million tonnes, out of which 40 percent is met from indigenous supply. A number of rock phosphate deposits have been recently identified in U.P., M.P., and Rajasthan. They are of different grades and require different methods of processing. Mining conditions also differ from place to place depending on the structure of the mineral deposits. Attempts are being made in India to explore large scale exploitation of indigenous rock phosphate.

0.3 In this standard sampling methods for rock phosphate have been outlined taking into consideration the current practices in the industry and also scientific principles of sampling. No attempt has been made to prescribe the procedures of sampling at the mining sites since this requires different approach altogether.

0.4 For the determination of size distribution of rock phosphate, sieves conforming to IS : 460 (Part 1)-1978* and IS : 460 (Part 3)-1978† shall be used. When such sieves are not available, other equivalent standard sieves as judged by the aperture may be used.

0.5 In reporting the results of a test or analysis, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960‡.

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*Specification for test sieves: Part 1 Wire cloth test sieves (*second revision*).

†Specification for test sieves: Part 3 Methods of examination of test sieves (*second revision*).

‡Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard lays down the procedure for collection and preparation of samples from a lot of rock phosphate in order to determine the size distribution, moisture content and chemical composition of the mineral in the lot. Sampling of rock phosphate from conveyor belt, trucks, wagons and also from stock piles have been described. This standard is primarily intended for manual sampling.

1.2 This standard also prescribes a method for reporting the quality of the lot sampled.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Sized — Rock phosphate in the size range 10-25 mm.

2.2 Fines — Rock phosphate with maximum size of 10 mm.

2.3 Consignment — A quantity of rock phosphate delivered at one time or under one order.

2.4 Lot — The quantity of rock phosphate indicated to be of the same grade/category offered for inspection at one time. Two or more lots shall be formed from a consignment if it consists of different grades/categories or if it is known to be originating from different sources.

2.5 Sublot — The quantity of the mineral in each of the portions into which a lot is divided for the purpose of facilitating sampling operations.

2.6 Increment — The quantity of mineral obtained by a sampling device at one time from a lot or sublot.

2.7 Gross Sample — Sample as collected from a sublot, that is the quantity of mineral consisting of the required number of increment taken from a sublot.

2.8 Size Sample — The sample taken for the determination of the size distribution of a lot or sublot.

2.9 Division Ratio — The ratio of the mass of the sample before division to the mass of divided sample.

2.10 Individual Laboratory Sample — The quantity of mineral obtained by reducing a gross sample following a specified procedure and intended for laboratory testing to estimate the chemical composition of the sublot.

2.11 **Composite Sample (for the lot)** — The quantity of rock phosphate obtained by mixing together equal or proportionate quantities of the material from each of the individual laboratory samples representing sublots into which a lot has been divided.

3. GENERAL GUIDELINES FOR SAMPLING

3.1 Before sampling every consignment is to be examined for the purpose of homogeneity in respect of grade, category and condition of supply. Further the total tonnage of a consignmnet should be ascertained in advance. If necessary, two or more lots shall be formed from a consignment on the basis of information available.

3.2 Sampling shall be carried out for each lot separately.

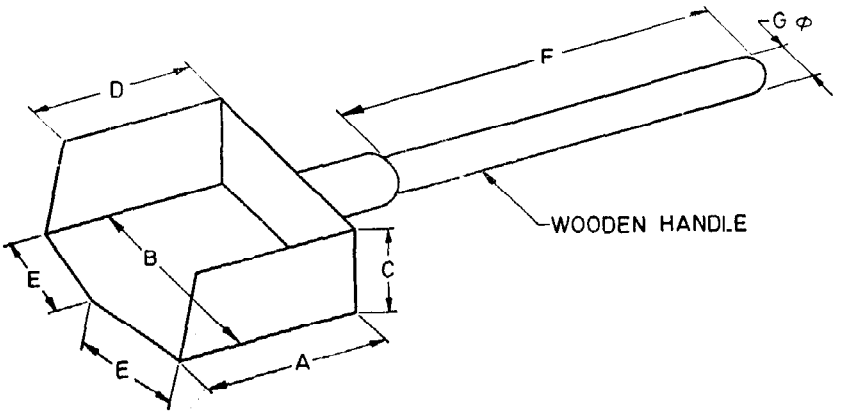
3.3 Sampling operations shall be carried out during shifting of the material at the time of loading, unloading, stockpiling or weighting in order to collect representative samples of the mineral. A lot is to be sub divided into a number of sublots on the basis of mass. This is done mainly to facilitate sampling operations and also to ensure that increments are drawn from all parts of the lot. If the lot is received or despatched continuously, sublots can be conveniently formed on the basis of the quantity moved during a fixed duration. However, the minimum number of sublots shall be as given in Table 1.

TABLE 1 MINIMUM NUMBER OF SUBLOTS

(*Clauses* 3.3, 5.1.1 *and* 5.3.2)

Mass of Rock Phosphate in Tonnes	Minimum Number of Sublots
Up to 1 000	1
1 001 to 3 000	2
3 001 to 5 000	3
5 001 to 10 000	5
10 001 and above	7

3.4 The increment sizes prescribed in Table 2 apply only to manual sampling. The samples are to be collected by using a suitable sampling scoop made of non corrosive metal or any other suitable device. An example of the sampling scoop is given in Fig. 1.



(All dimensions in millimetres.)

	A	B	C	D	E	F	G
2 kg	140	100	100	120	55	350	30
1 kg	140	100	50	120	55	350	30

FIG. 1 SAMPLING SCOOP

TABLE 2 MINIMUM NUMBER OF INCREMENTS AND MASS OF GROSS SAMPLE

(*Clauses* 3.4, 5.1.2, 5.2.2 *and* 5.3.2)

	SIZED	FINES
Mass of gross sample, in kg, *Min*	40	10
Mass of increment, in kg	2	1
Number of increments, *Min*	20	10

3.5 For each sublot there will be a corresponding gross sample. This gross sample shall be used for size determination and shall then be divided and reduced in stages to get a final laboratory sample.

3.6 Throughout the duration of sampling and sample preparation, the sample shall be protected from any alteration.

3.7 Gross samples as well as laboratary samples shall be stored in suitable bags or containers and marked with all details of sampling, such as sublot number, the date, the mass of the sample, etc.

4. MOISTURE SAMPLE

4.1 For each sublot moisture sample shall be collected separately. For this purpose minimum 5 increments shall be collected at equal intervals within a sublot and these shall be stored in an air-tight container with relevant details of sampling. When a sublot is handled on two or more days, moisture samples shall be collected for each day separately corresponding to the portions handled on the day.

5. SAMPLING PROCEDURE

5.1 Sampling from Conveyers

5.1.1 When a consignment of rock phosphate is received from a single source and moved on a conveyer to a destination such as factory site, storage yard, etc, the consignment may be considered as a lot for the purpose of sampling. Accordingly the number of sublots may be predetermined as per Table 1 with the knowledge of tonnage of the consignment. The division into the sublots may be done on the mass basis keeping equal intervals of mass.

5.1.2 Rock phosphate moved on a conveyer during one such interval shall form a sub-lot. A representative gross sample shall be drawn from each sublot. For this a required number of increments as per Table 2 shall be collected at the equal time intervals within each sublot.

5.1.3 When the material is in motion on a conveyer belt, the most reliable means of collecting increment is to sample at a point where it discharges from the belt or at a suitable transfer point in between. The sampling scoop as given in Fig. 1 may be used to collect increment. If this is not practicable due to fast flow of mineral, a suitable receptacle with wide opening to cover the falling stream may also be used.

5.2 Sampling from Wagons/Trucks

5.2.1 For the purpose of sampling, the wagons/trucks received under one order or in the course of one week may be termed as a lot provided the mineral is from the same source and consists of the same grade and category. For each lot sub division into sublots may be done according to Table 1.

5.2.2 If the number of wagons/trucks to be loaded or unloaded is known in advance, a random selection of 20 or 10 wagons/trucks shall be made depending on the size of material and from each of these one increment shall be collected when the material is in motion. However, if the number of wagons/trucks is not known in advance, one increment shall be drawn from each of the wagons/trucks at the site. This is done to ensure that minimum 20 increments are drawn as per Table 2 from each sublot in the case of sized mineral and 10 increments in the case of fines.

5.2.3 The increment shall be drawn by using sampling scoop or any other suitable sampling device when the material is loaded into or unloaded from the sample wagons/trucks.

5.3 Sampling from Stockpile

5.3.1 Sampling of rock phosphate in stationary stockpiles shall be avoided. The proper method of sampling is to collect the increment at the time of formation or dismantling of stockpiles.

5.3.2 It would be convenient to treat each stockpile as a lot or sublot depending on the tonnage as per Table 1. From each sub-lot, the number of increments to be taken shall be as per Table 2 and there shall be one gross sample for each sublot consisting of these increments. The increments shall be spaced at regular intervals at the time of formation or dismantling of stockpiles.

6. DETERMINATION OF SIZE DISTRIBUTION

6.1 Each gross sample shall be subjected to determination of size distribution separately. For this suitable IS sieves or equivalent may be used. The following sieves are recommended for sized and fines.

a) Sized : 22·4 mm, 10 mm and 2·80 mm

b) Fines : 10 mm, 2·80 mm, 1·40 mm, 850 μm, 250 μm, 150 μm and 53 μm.

6.2 Size Determination for the Lot — Each gross sample shall be weighed and its mass noted. The gross sample shall be screened through selected sieves and the mass retained on each of the sieves and the mass passing through the smallest sieves shall be noted separately. If m_1 is the mass of the gross sample pertaining to the first sublot, m_2 the mass of the gross sample pertaining to the second sublot, and so on, and a_1 is the mass of the mineral in a particular size range for the first sub lot, and a_2 the mass in that size range pertaining to the second sublot, then size fraction expressed as percentage for the size range is given by

$$\frac{a_1 + a_2}{m_1 + m_2} \times 100 \text{ for the lot.}$$

Similarly the size fraction of the lot for other size ranges may also be determined. The percentage shall be expressed correct to first decimal place.

7. REDUCTION OF GROSS SAMPLE

7.1 Each gross sample shall be reduced separately. Mineral of sizes greater than 22·4 mm shall be first crushed in a jew crusher, roll crusher or by manual method till the size – 22·4 mm is reached. The stages of successive reduction are shown in Fig. 2.

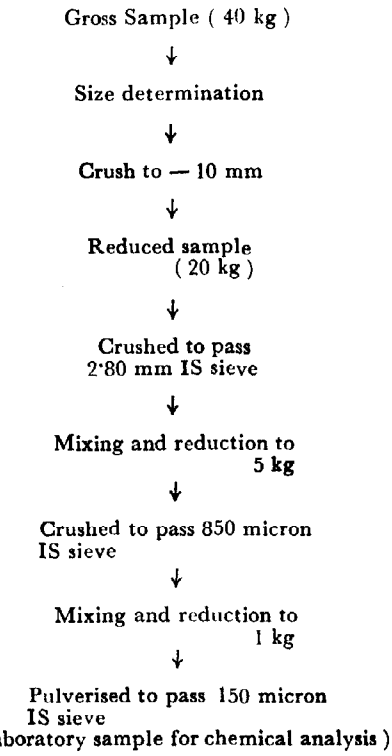


FIG. 2 STAGES OF SAMPLE PREPARATION

7.2 Moisture sample collected as per **4** shall be reduced separately. In the case of sized ores, the sample shall be crushed to — 10 mm and in the case of fines no crushing is required for moisture determination.

7.3 Sample Division Procedures — Sample division may be carried out either by riffle division or coning and quartering method if the reduction ratio is 2 : 1. For riffle division, riffles of suitable sizes shall be used. In the case of coning and quartering method, the material to be divided shall well be mixed and scooped into a cone-shaped pile. Care shall be taken to drop each scoopful exactly over the same spot in order to ensure even distribution of particles. After the cone is formed, it shall be flattened by pressing the top of the cone with the smooth surface of the scoop. Then it is cut into quarters by two lines which interesect at right angles at the centre of the cone. The bulk of the sample is reduced by rejecting two diagonally opposite quarters.

If a division ratio of more than 2 : 1 is desired the increment reduction method shall be used. For this purpose, the crushed material shall be spread on a smooth non-moisture absorbing plate into a uniform rectangle of even thickness. The length of the rectangle shall be divided into five equal parts, and the width into four equal parts so that it is divided into 20 equal cells. From each of the 20 portions a prescribed amount of material shall be taken with the help of a suitable scoop or spatula and then combined together to form a reduced sample of required mass.

7.4 Laboratory samples as well as moisture samples shall be obtained in duplicate for each gross sample. These samples shall be kept in a well stoppered container free from contamination.

8. NUMBER OF TESTS AND REPORTING OF TEST RESULTS

8.1 Individual laboratory samples shall be tested for total P_2O_5, Cao and SIO_2. For other characteristics a composite sample shall be prepared for each lot and tested. The composite sample shall be constituted by mixing equal or proportionate quantities of material from each of the individual laboratory samples.

8.2 The moisture results obtained from each sublot shall be individually reported. Similarly the results of individual laboratory samples (wherever analysed) shall be reported and the estimate for the lot as a whole shall be made as follows:

a) When only one laboratory sample is analysed, it shall be reported, as such; and

b) When two laboratory samples have been analysed individually and the results are x_1, x_2 the average of the two results shall be reported as the value of the characteristic for the lot sampled. However, if the sublots are of varying masses m_1 and m_2 the weighted average of the results shall be completed taking the sublot mass as weight that is $\overline{X} = \frac{m_1x_1 + m_2x_2}{m_1 + m_2}$

when three or more laboratory samples have been analysed individually from a lot for any characteristic, the following procedure shall be followed to assess the average quality and its limits of variation.

Let x_1, x_2, x_3 ....x_n be the results of analyzing 'n' laboratory samples for a particular characteristic.

Calculate

Average $(\overline{X}) = \frac{(x_1 + x_2 + \ldots\ldots\ldots x_n)}{n}$ If sublots are of approximately equal mass

or Weighted Average $(\overline{X}) = \frac{(m_1x_1 + m_2x_2 + \ldots\ldots\ldots m_nx_n)}{m_1 + m_2 + \ldots\ldots m_n}$

If m_1, m_2..... ...m_n are the masses of the sublot

and Range (R) = the difference between the maximum and minimum values among x_1,.....x_n

The average level of the characteristic in the lot shall be reported as equal to $\overline{X}$.

The limits of variation in the average level of the lot shall be reported as $\overline{X} + hR$ where h is a factor the value of which depends on the number of the sample analysed. The appropriate value of h may be taken from the following:

Number of Laboratory Samples Analysed (n)	*Value of Factor*, h
3	1·30
5	0·51
7	0·33

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